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I, KAY WARD, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. PP 6313 for a patent by M.I.M. HOLDINGS LIMITED filed on 06 October 1998.

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(ACN 009 814 019)

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Patents Act 1990

PROVISIONAL SPECIFICATION

Invention Title: METHOD FOR TREATING PRECIOUS METAL BEARING SULPHIDES

This invention is described in the following statement:

TITLE

METHOD FOR TREATING PRECIOUS METAL BEARING SULPHIDES

FIELD OF THE INVENTION

The present invention relates to a process for leaching refractory sulphide ores or concentrates and in particular is directed towards a method of recovering precious metals contained in the ores or concentrates.

BACKGROUND ART

Gold is generally extracted from gold containing ores by treatment with a cyanide solution which solubilizes the gold. However, in some ores, the gold is present as microparticles encapsulated within the ore. The gold in such ores cannot be extracted by traditional cyanidation techniques. These types of ores are known as refractory ores and are typically sulphide ores.

In order to extract gold from refractory ores, the ores must first be treated to liberate the gold so as to be accessible to cyanide leaching. A refractory ore is typically treated by oxidizing the ore which results in the chemical destruction of the sulphide matrix. Known methods of oxidising refractory ores include roasting and leaching the ore at elevated temperatures and pressure under acidic conditions. An example of such a process is known as the Sherritt process which includes the steps of feed preparation, pressure oxidation in the presence of acid and oxygen, solid/liquid separation, liquid neutralization and gold recovery from the oxidised solids by cyanidation. The operating conditions required in this process are at temperatures of between about 150°C to 210°C, a total pressure of 2,100 kPa, a pulp density of 20% to 30% solids by mass, acid concentration of 20 – 100 g/L and a retention time of two to three hours. The oxidation must be carried out in an autoclave and requires a source of oxygen. The capital costs associated with providing the autoclaves and meeting a high oxygen demand are high and may be prohibitive for construction at remote sites, for medium to small scale

operations and for low grade ore. It is possible to leach under less aggressive conditions but in this case, the leaching rates and recovery are too low to be economically viable.

Attempts have been made to reduce the aggressive conditions and to lower the pressures and temperatures required whilst maintaining the economic viability of the process. For example, Australian patent application no. 27182/92 describes subjecting an ore concentrate to fine grinding prior to leaching. Fine grinding to an 80% passing size of 15 micron or less enabled the leaching to be carried out under less aggressive conditions at temperatures of 95-110°C and pressures of about 1000 kPa.

Thus, whilst some progress has been made in reducing the operating parameters when using oxygen as the oxidant, the leach must still be carried out under pressure.

It is also known to oxidatively leach mineral species with ferric ions under acidic conditions. Ferric ion is typically a more effective oxidizing agent than oxygen which means that oxidation with ferric ions can be carried out under less aggressive conditions. Oxidative leaching at atmospheric pressure using ferric ions is known. A disadvantage of leaching with ferric ions is that the ferric ions are reduced to ferrous ions during the leaching reaction. An increase in ferrous ion concentration adversely affects the rate of leaching. Thus, it is necessary to remove the ferrous ion during the leach. Further, as leaching solutions are recycled, ferric ions must be regenerated by oxidizing the reduced ferrous.

A common disadvantage with the aforementioned processes is that they operate under acidic conditions. As cyanidation requires alkaline conditions, the acid must be removed prior to cyanidation. This may be overcome by leaching under alkaline conditions and such leaching is known. However, a major disadvantage of

alkaline leaching is that when iron containing ores such as pyrite are oxidised, the sulphide iron which is leached precipitates as a passive iron oxide layer on the mineral particle. This layer inhibits further oxidation with the result being that the extent of leaching under alkaline conditions is less than under acidic conditions. This translates to a lower recovery of precious metals.

Still further, alkaline leaching also requires elevated pressure and temperatures and an oxygen source for the leaching to occur at a rate and provide gold recoveries which may be economically viable. However, even under aggressive conditions, recovery of precious metals is less than that for acid leaching.

Further, base metals such as copper and zinc are insoluble at high pH. Thus, alkaline leaching is unsuitable for leaching ores or concentrates where recovery of base metals from base metal sulphides such as chalcocite, sphalerite or chalcopyrite is required. For these reasons, commercial and academic interest has been directed towards acid leaching.

Most of the literature relating to alkaline leaching is directed towards the use of alkalis such as sodium or potassium hydroxide and ammonia. A disadvantage with these reagents is that iron is precipitated primarily as jarosite. Jarosite inhibits gold recovery and is also an environmentally unacceptable residue. Also, hydroxide reagents and in particular sodium hydroxide are prohibitively expensive.

The use of cheaper alkalis such as lime has been proposed. However, to date, leaching of iron sulphide materials with lime has been unsuccessful in that leaching is incomplete and subsequent precious metal recovery is low. For example, an earlier study of alkaline oxidation of pyrite in gold recovery using lime achieved only 30 to 40% gold recovery which offered little improvement over direct cyanidation of the pyrite. This is believed to be due to passivation of the mineral by precipitation of an alkali jarosite/gypsum/iron oxide

layer. Limestone is another alkali which is relatively cheap. Limestone is typically used in the neutralization of acidic leachates. However, limestone is considered to be insufficiently reactive and/or soluble in alkaline systems to be able to be used for alkaline leaching.

From an economic point of view, it would be desirable to be able to leach refractory materials for precious metal recovery under mild alkaline conditions and using reagents other than the expensive hydroxides. As mentioned above, it is known that the oxidation rate under acidic conditions can be increased by fine grinding to increase the surface area of the mineral particles. Such an increase may be predicted given that there is a larger surface area exposed to the oxidizing agents. However in the alkaline system, this is not the case in view of the formation of the passive iron oxide layer on the particles. The rate determining factors in the alkaline systems rates are believed to relate to the formation of the passivating iron oxide layer and diffusion of oxygen through the layer. Thus, workers in the field have concentrated on increasing the extent of alkaline leaching by using strong, soluble alkalis, by modifying the leaching conditions so as to minimise formation of the passive layer and/or influence the diffusion rate through the layer. One study suggests leaching at higher temperatures or at relatively concentrated solutions of reagents. The reason for this is to rapidly produce a passive layer which is unstable and subject to cracking. It is believed that at lower temperatures, the layers grow more slowly and are more stable. Another suggestion has been to use additives which may react to dissolve the layer or to make the layer more permeable.

However, to date no method proposed has been proposed which is able to successfully leach iron containing refractory ores under alkaline conditions and which also enables good recovery of precious metals from the ore.

The present invention is based on the surprising and unexpected discovery that leaching an iron containing refractory material under alkaline conditions can be successfully achieved by careful selection of the particle size of the material to be leached. Even more surprising it has been discovered that not only can the leaching efficiency be improved but that leaching can also be successfully conducted under relatively mild conditions.

According to a first broad form of the invention there is provided a method of processing a mineral composition comprising an iron containing refractory sulphide, the method comprising milling the composition to a particle size of P_{80} of less than 25 microns and leaching said composition with a solution comprising lime and/or limestone in the presence of an oxygen containing gas.

The method of the present invention is applicable to a composition including an iron containing refractory sulphide material or a mixture of such materials. Examples of such materials include pyrite, marcasite, arsenopyrite, troilite and pyrrhotite. Other non-iron or minor iron containing materials may also be present in the composition, examples of which include stibnite, tetrahedrite, tennantite and pentlandite. Suitably the composition would not include economic amounts of base metal sulphides containing copper or zinc. Generally a composition which includes appreciable amounts of these base metals would not be treated directly by the method of the present invention. The reason for this is purely economical as copper and zinc precipitate under alkaline conditions and thus cannot be recovered easily. Under acidic leaching conditions, copper and zine are solubilized and can be recovered by conventional SX/EW techniques. Use of flotation or other separation technologies to produce a base metal concentrate and a separate refractory iron sulphide concentrate would be obvious to one skilled in the art as

a way of treating these materials.

The method of the present invention is in particular directed towards the treatment of refractory materials containing precious metals such as gold, silver and platinum. Suitably, the refractory materials are in the form of ores flotation concentrates although the method is suitable for ores if the economics are favourable.

In the method of the present invention, the composition is finely ground to a particle size of P_{80} less than 25μm. A typical particle size range is between 5-25μm and preferably between about 12-20μm.

A preferred apparatus for producing the finely ground material is a stirred mill. However, it will be appreciated that other types of comminution apparatus may also be used such as wet and dry vibratory mills or planetary mills.

The leaching is carried out using lime and/or limestone as the alkali reagent. Lime, limestone or a mixture thereof may be used. Preferably a mixture in the range of 40-95% limestone is used. The lime and/or limestone is added in an amount such that the pH of the system is between about 7-12 and preferably about 8-10.

The present inventors have also surprisingly discovered that not only can the overall amount of leaching be increased but that such an increase can also be obtained by leaching under milder conditions than has hitherto been possible. Suitably the method of the present invention can be carried out at ambient pressure. This avoids the use of expensive pressure reactors and autoclave equipment. The preferred operating temperature is between about 50°C up to the boiling point of the mixture. Typically the maximum temperature is about 95°C.

The leaching reaction can thus be carried out in open tank reactors. Excess heat is removed by evaporation of the solution. This avoids the need for costly heat exchangers. Heat can be easily introduced by

known methods such as the injection of steam.

The leaching reaction is carried out in the presence of an oxygen containing gas. When the reaction is carried out in an open tank reactor the gas is typically introduced by sparging. The gas may be oxygen, air or oxygen enriched air. The gas flow is dependent upon the amount of oxygen required to sustain the leaching reaction and is typically about 0.01 – 0.5 vvm.

After the composition has been leached, the mixture can be further treated by known methods to recover precious metals, principally by cyanide leaching.

Cyanidation occurs under alkaline conditions. As the leach solution is already alkaline, the solution may be subjected directly to cyanide leaching. If desired the slurry may be thickened prior to cyanide leaching.

According to a second broad form of the invention there is provided a method of recovering precious metals from a mineral composition comprising an iron containing refractory sulphide material, the method comprising;

grinding the material to a particle size P_{80} of at least 25μm;

leaching the ground material in the presence of lime and/or limestone and an oxygen containing gas; and

subjecting the leached material to a further leaching step to recover any precious metals.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a flow diagram of a preferred method of the present invention and Figure 2 is a flow diagram of a further preferred method of the present invention.

BEST MODE

The present invention will now be described in relation to the following examples. However, it will be appreciated that the generality of the invention as described above is not limited to the following examples.

Example No. 1. Whole ore leach of a pyrite containing ore, with a 50:50 blend of limestone and lime, oxygen used as the oxidant.

Alkali Leach

The ore sample was slurried in tap water at 50% solids and ground in a laboratory rod mill for a period of 20 minutes to achieve a slurry ground to 80% passing 106 microns. The slurry was then thickened to 60% solids, and milled in a Netzsch horizontally stirred laboratory scale bead mill. The media used was 0.8 – 1.1mm steel balls. Slurry was passed through the mill twice to achieve the required grind size of 80% passing 14 microns. The final particle size distribution was determined by lasersizer.

The leaching test was carried out in a cylindrical stainless steel reactor fitted with four baffles located equidistant around the edges of the vessel. A 1,000 gram sample of the ground solids were added to the leach vessel along with 20 litres of tap water. The live volume of the reactor was 25 litres. The vessel was fitted with a stainless steel lid to prevent evaporative loss from the reactor. The slurry was agitated by a 6 bladed Rushton style impeller. Oxygen was introduced to the reactor by an air spear, which terminated below the impeller. The oxygen flow was controlled by a rotameter at 2,000 cubic centimetres per minute (0.1 vvm).

The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The limestone/lime slurry consisted of 50% lime and 50% limestone, at a slurry density of 35% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The

controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was heated by steam injection into a jacket surrounding the vessel. The steam injection was regulated by a solenoid valve, which was controlled by an automatic temperature controller. The vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the filter cake was analysed for residual sulphide by acid evolution. A further sample of the cake was sent for XRD analysis to confirm the sulphide analysis. The dry weight of the filter cake was 1150 grams. The results of the sulphide oxidation step are listed below in Table 1.

Table 1

Degree of sulphide Oxidation Achieved for Whole Ore Leach

Sample	Weight - grams	% sulphide sulphur	% Pyrite by sulphide evolution	% Pyrite by XRD	% sulphide oxidation
Feed	1000	8.2	15.2	15.8	90.1
Leach Residue	1150	0.7	1.30	1.6	

Example No. 2. Leach of a pyrite containing concentrate, with a 50:50 blend of limestone and lime, oxygen used as the oxidant.

Alkali Leach

The concentrate sample was slurried in tap water at 60% solids, and milled in a Netzsch horizontally

stirred laboratory scale bead mill. The media used was 0.8 – 1.1mm steel balls. Slurry was passed through the mill twice to achieve the required grind size of 80% passing 12 microns. The final particle size distribution was determined by lasersizer.

The leaching test was carried out in a cylindrical stainless steel reactor fitted with four baffles located equidistant around the edges of the vessel. A 200 gram sample of the ground solids were added to the leach vessel along with 2000mL of tap water. The live volume of the reactor was 2.5 litres. The vessel was fitted with a stainless steel lid to prevent evaporative loss from the reactor. The slurry was agitated by a 6 bladed Rushton style impeller. Oxygen was introduced to the reactor by an air spear, which terminated below the impeller. The oxygen flow was controlled by a rotameter at 200 cubic centimetres per minute (0.1 vvm).

The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The limestone/lime slurry consisted of 50% lime and 50% limestone, at a slurry density of 35% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was placed on a thermocouple controlled hotplate, with the thermocouple immersed in the slurry through a stainless steel sheath. The vessel temperature was maintained at the required temperature by the thermocouple controller. The outside of the vessel was wrapped in insulated foam to prevent heat loss. The

vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the filter cake was analysed for residual sulphide by acid evolution. A further sample of the cake was sent for XRD analysis to confirm the sulphide analysis. The dry weight of the filter cake was 433 grams. The results of the sulphide oxidation step are listed below in Table 2.

Table 2

Degree of Sulphide Oxidation Achieved for Pyrite Concentrate

Sample	Weight – grams	% sulphide sulphur	% Pyrite by acid evolution	% Pyrite by XRD	% sulphide oxidation
Feed	200	33.2	61.73	64	91.5
Leach Residue	430	1.3	2.41	3.0	

Cyanide Leach

A sample of the leach residue was slurried in tap water at a slurry density of 40% w/w solids in a baffled glass reactor. The volume of the reactor was 3 litres. The slurry was agitated by a 6 bladed Rushton style impeller. Air was introduced to the reactor by an air spear, which terminated below the impeller.

The pH of the slurry was adjusted and maintained at pH 10 using lime, and sodium cyanide was added to the slurry to maintain a free cyanide level of 500ppm. The test was carried out for a period of 24 hours. On completion of the test, the slurry was filtered and the filter cake washed with deionised water. The filtrate and washings were combined for analysis. The solid filter cake was dried in an oven and analysed to

determine the precious metals recovery.

The results of the cyanide leach are listed in Table 3.

Table 3.
Cyanide Leach Results for Oxidised Pyrite Concentrate

Sample	% pyrite oxidised	Gold Extraction %
Feed prior to oxidation	NA	26.6
Oxidised Residue	90.1	97.4

The precious metals extractions were determined by head and tails fire assay.

Example No. 3. Leach of a pyrite containing concentrate, with a 50:50 blend of limestone and lime, air used as the oxidant.

Alkali Leach

The concentrate sample was slurried in tap water at 60% solids, and milled in a Netzsch horizontally stirred laboratory scale bead mill. The media used was 0.8 – 1.1mm steel balls. Slurry was passed through the mill twice to achieve the required grind size of 80% passing 12 microns. The final particle size distribution was determined by lasersizer.

The leaching test was carried out in a cylindrical stainless steel reactor fitted with four baffles located equidistant around the edges of the vessel. A 200 gram sample of the ground solids were added to the leach vessel along with 2,000mL of tap water. The live volume of the reactor was 2.5 litres.

The vessel was fitted with a stainless steel lid to prevent evaporative loss from the reactor. The slurry was agitated by a 6 bladed Rushton style impeller. Air was introduced to the reactor by an air spear, which terminated below the impeller. The air flow was controlled by a rotameter at 200 cubic centimetres per minute (0.1 vvm).

The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The limestone/lime slurry consisted of 50% lime and 50% limestone, at a slurry density of 35% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was placed on a thermocouple controlled hotplate, with the thermocouple immersed in the slurry through a stainless steel sheath. The vessel temperature was maintained at the required temperature by the thermocouple controller. The outside of the vessel was wrapped in insulated foam to prevent heat loss. The vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the filter cake was analysed for residual sulphide by acid evolution. The dry weight of the filter cake was 469 grams. The results of the sulphide oxidation step are listed below in Table 4.

Table 4

Degree of Sulphide Oxidation Achiev d for Pyrite Concentrate when oxidised with Air as the Oxidant

Sample	Weight – grams	% sulphide sulphur	% Pyrite by acid evolution	% sulphide oxidation
Feed	200	33.2	61.73	90.1
Leach Residue	469	1.4	2.6	

Example No. 4. Leach of an arsenopyrite containing concentrate, with a 50:50 blend of limestone and lime, oxygen used as the oxidant.

Alkali Leach

The concentrate sample was slurried in tap water at 60% solids, and milled in a Netzsch horizontally stirred laboratory scale bead mill. The media used was 0.8 – 1.1mm steel balls. Slurry was passed through the mill twice to achieve the required grind size of 80% passing 14 microns. The final particle size distribution was determined by lasersizer.

The leaching test was carried out in a cylindrical stainless steel reactor fitted with four baffles located equidistant around the edges of the vessel. A 1500 gram sample of the ground solids were added to the leach vessel along with 20L of tap water. The live volume of the reactor was 25 litres. The vessel was fitted with a stainless steel lid to prevent evaporative loss from the reactor. The slurry was agitated by a 6 bladed Rushton style impeller. Oxygen was introduced to the reactor by an air spear, which terminated below the impeller. The oxygen flow was controlled by a rotameter at 2000 cubic centimetres per minute (0.1 vvm).

The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of

10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The limestone/lime slurry consisted of 50% lime and 50% limestone, at a slurry density of 30% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was heated by steam injection into a jacket surrounding the vessel. The steam injection was regulated by a solenoid valve which was controlled by an automatic temperature controller. The vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the filter cake was analysed for residual sulphide by acid evolution. A further sample of the cake was sent for XRD analysis to confirm the sulphide analysis. The dry weight of the filter cake was 2965 grams. The results of the sulphide oxidation step are listed below in Table 5.

Table 5

Degree of Sulphide Oxidation Achieved for Arsenopyrite Concentrate

Sample	Weight grams	% sulphide sulphur	% Arsenopyrite by XRD	% Pyrite by XRD	% sulphide oxidation
Feed	1500	20.1	30.6	29.5	93.1
Leach Residue	2965	0.7	<1	1.4	

The presence of arsenic as ferric arsenate in the leach

residue was confirmed by XRD

Cyanide Leach

A sample of the leach residue was slurried in tap water at a slurry density of 40% w/w solids in a baffled glass reactor. The volume of the reactor was 3 litres. The slurry was agitated by a 6 bladed Rushton style impeller. Air was introduced to the reactor by an air spear, which terminated below the impeller.

The pH of the slurry was adjusted and maintained at pH 10 using lime, and sodium cyanide was added to the slurry to maintain a free cyanide level of 500ppm. The test was carried out for a period of 24 hours. On completion of the test, the slurry was filtered and the filter cake washed with deionised water. The filtrate and washings were combined for analysis. The solid filter cake was dried in an oven and analysed to determine the precious metals recovery.

The results of the cyanide leach are listed in Table 6.

Table 6.
Cyanide Leach Results for Oxidised Pyrite and Arsenopyrite Concentrate

Sample	% pyrite oxidised	% arsenopyrite oxidised	Gold Extraction %
Feed	NA	NA	35.8
Oxidised Residue	90.6	>95	88.05

The precious metals extractions were determined by head and tails fire assay.

Example No. 5. The Effects of Varying Alkali Mixtures on the Extent of Sulphide Oxidation

Alkali Leach

Approximately 5 kg of pyrite concentrate sample was slurried in tap water at 60 % solids, and milled in a Netzsch horizontally stirred laboratory scale bead mill. The media used was 0.8 – 1.1 mm steel balls. Slurry was passed through the mill twice to achieve the required grind size of 80% passing 8.6 microns. The final particle size distribution was determined by lasersizer. On completion of the grind, the ground slurry was split into 500 gram subsamples for the leaching test, and each subsample filtered separately. All subsamples were stored frozen to prevent oxidation of the pyrite.

A 200 gram sample was slit out of each subsample for the leaching testwork.

The leaching test was carried out in a cylindrical stainless steel reactor fitted with four baffles located equidistant around the edges of the vessel. A 200 gram sample of the ground solids were added to the leach vessel along with 2 litres of tap water. The live volume of the reactor was 2.5 litres. The vessel was fitted with a stainless steel lid to prevent evaporative loss from the reactor. The slurry was agitated by a 6 bladed Rushton style impeller. Oxygen was introduced to the reactor by an air spear, which terminated below the impeller. The oxygen flow was controlled by a rotameter at 400 cubic centimetres per minute (0.2 vvm).

The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The limestone/lime slurry composition was varied for the three leach tests according to the following ratios: 100% lime, 50% lime 50% limestone, 10% lime 90% limestone.

For all tests, the slurry density of the alkali

blend was of 35% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was placed on a thermocouple controlled hotplate, with the thermocouple immersed in the slurry through a stainless steel sheath. The vessel temperature was maintained at the required temperature by the thermocouple controller. The outside of the vessel was wrapped in insulated foam to prevent heat loss. The vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the cake was sent for XRD analysis to determine the extent of pyrite leaching. The dry weight of the filter cake was recorded.

The results are listed in Table 7, below

Table 7.

The Effects Of Varying Lime/Limestone Blends On The Extent Of Pyrite Oxidation From Pyrite Flotation Concentrate

Alkaline Leach Tests				
	pH	10	10	10
	Alkali mixture	100% lime	50% lime 50% limestone	10% lime 90% limestone
	Grind Size - 80% passing	8.6	8.6	8.6
	% solids	10.0	10.0	10.0
Feed data:				
	Dry solids wt g	200	201.0	201.1

	% pyrite in head	65.0	65.0	65.0
Residue data:				
	% pyrite in residue	3.2	3.0	1.0
	% pyrite oxidised from XRD	95.47	90.1	94.3

Example No. 6. The Effects of Varying Grind Size on the Extent of Sulphide Oxidation.

Alkali Leach

Approximately 4 kg of pyrite concentrate sample was slurried in tap water at 60% solids, and milled in a Netzsch horizontally stirred laboratory scale bead mill. The media used was 0.8 – 1.1 mm steel balls. Slurry was passed through the mill in five consecutive passes, with a subsample taken from the ground slurry from each pass. Each subsample therefore was ground to a different particle size. The particle sizes from each pass were (80 % of mass passing):

Pass1	51.9 microns
Pass2	32.04 microns
Pass3	17.94 microns
Pass4	13.64 microns
Pass5	11.71 microns
Pass6	8.6 microns

The final particle size distribution was determined by lasersizer. On completion of the grind, the ground slurry was split into 500 gram subsamples for the leaching test, and each subsample filtered separately. All subsamples were stored frozen to prevent oxidation of the pyrite.

A 200 gram sample was slit out of each subsample for the leaching testwork.

The leaching test was carried out in a

cylindrical stainless steel reactor fitted with four baffles located equidistant around the edges of the vessel. A 200 gram sample of the ground solids were added to the leach vessel along with 2 litres of tap water. The live volume of the reactor was 2.5 litres. The vessel was fitted with a stainless steel lid to prevent evaporative loss from the reactor. The slurry was agitated by a 6 bladed Rushton style impeller. Oxygen was introduced to the reactor by an air spear, which terminated below the impeller. The oxygen flow was controlled by a rotameter at 400 cubic centimetres per minute (0.2 vvm).

The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The lime slurry was of 35% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was placed on a thermocouple controlled hotplate, with the thermocouple immersed in the slurry through a stainless steel sheath. The vessel temperature was maintained at the required temperature by the thermocouple controller. The outside of the vessel was wrapped in insulated foam to prevent heat loss. The vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the cake was sent for XRD analysis to determine the extent of pyrite leaching. The dry weight of the filter cake was recorded.

The results are listed in Table 8, below

Table 8.

The Effects of Varying Grind Size on the Extent of Pyrite Oxidation from Pyrite Flotation Concentrate

Alkaline Leach Tests						
pH	10	10	10	10	10	10
Alkali mixture	100% lime	100% lime	100% lime	100% lime	100% lime	100% lime
Grind Size - 80% passing	51.9	32.04	17.94	13.64	11.71	8.6
% solids	10.0	10.0	10.0	10.0	10	10.0
Feed data:						
Dry solids wt g	200.0	200	200	200	200	200
% pyrite in head	65.0	65.0	65.0	65.0	65.0	65.0
Residue data:						
% pyrite in residue	16.7	9.7	7	2.7	3.7	3.2
% pyrite oxidised from XRD	73.9	80.1	91.06	96.81	95.54	95.47

Example No. 7. Leach of a pyrite containing concentrate, with a 50:50 blend of limestone and lime with oxygen used as the oxidant, at varying pH.

Alkali Leach

The concentrate samples were slurried in tap water at 60% solids, and milled in a Netzsch horizontally stirred laboratory scale bead mill. The media used was 0.8 – 1.1mm steel balls. Slurry was passed through the mill twice to achieve the required grind size of 80% passing 12 microns. The final particle size distribution was determined by lasersizer.

All leaching tests were carried out in a

cylindrical stainless steel reactor fitted with four baffles located equidistant around the edges of the vessel. A 2000 gram sample of the ground solids was added to the leach vessel along with 20 L of tap water. The live volume of the reactor was 25 litres. The vessel was fitted with a PVC lid to prevent evaporative loss from the reactor. The slurry was agitated by a 6 bladed Rushton style impeller. Oxygen was introduced to the reactor by an air spear, which terminated below the impeller. The oxygen flow was controlled by a rotameter at 1500 cubic centimetres per minute (0.075 vvm).

The pH in the vessel was maintained by an automated pH analyser and controller. In all, three tests were carried out, each at a different pH. The pH control points used were

Test 1	pH 8
Test 2	pH 9
Test 3	pH 10

A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The limestone/lime slurry consisted of 50% lime and 50% limestone, at a slurry density of 35% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was heated by steam injection into a jacket surrounding the vessel. The steam injection was regulated by a solenoid valve which was controlled by an automatic temperature controller. The vessel temperature was controlled to 85°C. The outside of the vessel was

wrapped in insulated foam to prevent heat loss.

On completion of the test, each slurry was filtered and the filter cake dried and weighed.

Cyanide Leach

A sample of each leach residue was then slurried in tap water at a slurry density of 40% w/w solids in a baffled glass reactor. The volume of the reactor was 3 litres. The slurry was agitated by a 6 bladed Rushton style impeller. Air was introduced to the reactor by an air spear, which terminated below the impeller.

The pH of the slurry was adjusted and maintained at pH 10 using lime, and sodium cyanide was added to the slurry to maintain a free cyanide level of 500ppm. The test was carried out for a period of 24 hours. On completion of the test, the slurry was filtered and the filter cake washed with deionised water. The filtrate and washings were combined for analysis. The solid filter cake was dried in an oven and analysed to determine the precious metals recovery.

Table 9.

Gold Recoveries from Oxidised Pyrite concentrate at Varying pH

Control pH	Not oxidised	Oxidised at pH 8	Oxidised at pH 9	Oxidised at pH 10
Au recovery - %	26.6	92.2	96.1	97.2

The precious metals extractions were determined by head and tails fire assay.

It can be seen that by grinding 25μm or less 90% or more of the pyrite is oxidised. A high level of pyrite oxidation typically translates to high precious metal recovery.

The methods of the present invention offer a number of advantages over existing methods. First, iron containing refractory ores such as pyrite can be leached to high levels under alkaline conditions using lime and/or limestone as the alkali source. Lime and limestone offer significant economic advantages over the known agents such as caustic. For example, current costs for caustic are about AUD$440 per tonne, ammonium based salts, about AUD$450, lime AUD$100-200 and limestone AUD$15-41/tonne.

Further, the leach need not be pressurised which translates to significant capital and operating costs. Still further, costs can be reduced as the leach can be carried out using air instead of oxygen. This means that oxygen need not be purchased or produced.

The leach residence times for the preferred methods of the present invention are typically about a few hours.

These residence times compare favourably to oxidative leaching under acidic conditions and are in fact superior to atmospheric ferric leaching.

During the leaching iron precipitates as goethite and hematite, rather than Jarosite as occurs under acidic conditions. Jarosite inhibits subsequent precious metal recovery by making the residue difficult to settle and filter. Further, Jarosite is not an environmentally acceptable residue.

Gypsum is also formed during the leach and precipitates. An advantage of gypsum is that it enhances the filterability of the residue. Gypsum is not formed when using conventional alkali reagents.

Another advantage over the use of the conventional alkali reagents is that in the present

invention arsenic present in the refractory material is precipitated as ferric arsenate. The level of arsenic in the leach liquor is typically below detectable limits. When using conventional alkalis arsenic is present in the leach liquor.

DATED this Sixth day of October 1998

M.I.M. HOLDINGS LIMITED

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